

Claims:

An integrated process for preparation of sulphate of potash (SOP) from bittern comprising:

- (i) subjecting bittern to fractional crystallization to obtain kainite type mixed salt with high kainite content and MgCl₂-rich end bittern, and subjecting the MgCl₂ rich end bittern to desulphation;
 - 5 (ii) treating the kainite type mixed salt with water and mother liquor obtained in step (xiii) below to leach out substantially all NaCl from the mixed salt and simultaneously convert kainite into schoenite;
 - 10 (iii) filtering the schoenite and separating the filtrate;
 - (iv) desulphating the filtrate with aqueous CaCl₂;
 - (v) filtering the gypsum produced in step (iv) and mixing the filtrate with the MgCl₂-rich filtrate obtained in step (vii) below;
 - 15 (vi) evaporating the resultant solution of step (v) and cooling to ambient temperature to crystallize crude carnallite;
 - (vii) centrifuging the crude carnallite and recycling the required quantity of filtrate to step (v);
 - (viii) decomposing the crude carnallite with appropriate quantity of water from step (vi) to yield crude KCl and carnallite decomposed liquor;
 - 20 (ix) filtering the crude KCl, and washing with water to remove adhering MgCl₂ and subjecting to hot leaching for production of MOP and NaCl;
 - (x) mixing the carnallite decomposed liquor from step (viii) and washing from step (ix) and treating with hydrated lime;
 - (xi) filtering the slurry and washing the cake to produce Mg(OH)₂ and CaCl₂-containing filtrate for the desulphation process of step (iv);
 - 25 (xii) treating by known method the schoenite produced in step (iii) with MOP produced in step (ix) to produce SOP under ambient condition;
 - (xiii) filtering the SOP and collecting separately the mother liquor hereinafter referred to as KEL;
 - 30 (xiv) recycling the KEL of step (xiii) in the process of step (ii).
2. A process as claimed in Claim 1 wherein the bittern contains K, Mg and SO₄ in concentrations that make it suitable for kainite production
 3. A process as claimed in claim 2 wherein said bittern is selected from sea bittern and sub-soil bittern, and preferably bittern with higher potassium content and which

- also requires the least evaporation to produce kainite-type mixed salt, and waste bittern sources such as debrominated bittern effluent.
4. A process as claimed in any one of claims 1 to 3 wherein said mixed salt contains KCl-15-22%, NaCl-15-22%, MgSO₄-28-40%, MgCl₂-5-10%.
 5. A process as claimed in any preceding claim wherein one part by weight of mixed salt is treated with 0.75-1.25 parts by volume of KEL and 0.3-0.7 parts by volume of water.
 6. A process as claimed in any preceding claim wherein said KEL contains typically 15-17% KCl, 1-3% NaCl, 10-12% MgSO₄, and 2-3% MgCl₂.
 7. A process as claimed in any preceding claim wherein the SEL contains typically 8-10% KCl, 6-12% NaCl, 12-14% MgSO₄ and 5-7% MgCl₂.
 8. A process as claimed in any preceding claim wherein the schoenite contains typically 40-45% K₂SO₄, 30-35% MgSO₄ and 0.5-2.0% NaCl.
 9. A process as claimed in any preceding claim wherein the stoichiometric ratio of CaCl₂ to sulphate for the desulphatation reaction of step (iv) is 1.1:1 to 0.9:1, preferably 1:1.
 10. A process as claimed in claim 7 wherein 1 part by volume of desulphated SEL was mixed with 0.5-1.5 parts by volume of MgCl₂-rich end bittern of 36-38°Be' (s.g. 1.33-1.38), preferably, 0.7-0.9 parts of end bittern of 37 °Be' (sp. gr. 1.342), and more preferably, MgCl₂-rich end bittern containing no sulphate.
 11. A process as claimed in any one of claims 1 to 10 wherein the concentration of desulphated SEL to produce carnallite is carried out in a solar pan or in a multiple effect evaporator with simultaneous recovery of water.
 12. A process as claimed in any preceding claim wherein evaporation is continued till the solution attained a temperature in the range of 120-128°C and more preferably, 122-124°C.
 13. A process claimed in any preceding claim wherein the carnallite obtained has 15-20% KCl, 15-20% NaCl and 28-32% MgCl₂.
 14. A process claimed in any preceding claim wherein one part by weight of the carnallite is decomposed with 0.4-0.6 parts by volume of water, followed by washing of the cake with a small quantity of water.
 15. A process claimed in any preceding claim wherein the molar ratio of active lime to MgCl₂ for production of Mg(OH)₂ and CaCl₂ is in the range of 0.8-1.0, preferably, 0.90.

16. A process claimed in any preceding claim wherein said Mg(OH)₂ obtained is calcined to produce MgO with 94-98 % purity and with 0.02-0.04 % B₂O₃.
17. A process claimed in any preceding claim wherein the Mg(OH)₂ is used without upgradation for neutralization of acidified de-brominated bittern where such bittern is used as potash source.
18. A process claimed in any preceding claim wherein the MOP obtained upon hot leaching of crude KCl has a purity in the range of 92-98% and NaCl content of 1-5 %, preferably >95% KCl and <2% NaCl.
19. A process as claimed in claims 1-3 wherein the NaCl obtained on hot leaching of crude KCl contains > 97% NaCl.
20. A process claimed in any preceding claim wherein one part by weight of schoenite is treated with 0.3-0.6 parts by weight of MOP and 1-2 parts by volume of water, and more preferably 0.4 parts by weight of MOP and 1.5 parts by volume of water, in the ambient temperature range of 20-45 degree C.
- 15 21. A process claimed in any preceding claim wherein the SOP produced has K₂O content in the range of 50-52% and chloride in the range of 0.5-2.0 %.